

Досліджено фізико-хімічні особливості хром-нікельвмісних відходів виробництва корозійностійких сталей та легуючого сплаву, отриманого за допомогою відновлювальної плавки. Це необхідно для визначення параметрів, що знижують втрати Ni та Cr при переробці оксидної легованої сировини та використанні отриманої легуючої добавки. Визначено, що в сплаві при співвідношенні O/C в шихті в межах 1,09–1,78 присутні фази  $\gamma$ -Fe та  $Fe_3C$  з легуючими елементами в якості атомів заміщення. При O/C=1,78 фазовий склад переважно складався з  $\gamma$ -Fe при слабкому прояві  $Fe_3C$ . Поетапна зміна O/C в шихті на 1,33 та 1,09 призводила до підвищення прояву  $Fe_3C$  на дифрактограмах. Мікроструктура суміші окалини хром-нікельвмісних корозійностійких сталей розупорядкована з присутністю часток різного розміру та форми. Вміст легуючих елементів Ni та Cr складав 7,65 % мас. та 14,26 % мас. відповідно при вмісті кисню на рівні 29,70 % мас. Мікроструктура легуючого сплаву з різним співвідношенням O/C в шихті мала чіткий прояв декількох фаз, що характеризувалися різницею у вмісті основних легуючих елементів. Вміст Ni у досліджених ділянках різних фаз змінювався в межах 1,41–20,90 % мас., Cr – 1,27–32,90 % мас. Згідно досліджень найбільш прийнятним співвідношенням O/C в шихті є 1,78. В цьому випадку забезпечувалося відновлення з переважанням у фазовому складі  $\gamma$ -Fe з відносно слабким проявом залишкового вуглецю, як карбідної складової. Тобто визначено показники переробки хром-нікельвмісних техногенних відходів та отримання легованого продукту відновної плавки з відносно низьким вмістом вуглецю. Це розширює можливість ресурсозбереження з використанням отриманого сплаву з заміною деякої частини стандартних легуючих матеріалів при виплавці обмежених по вуглецю марок сталей

**Ключові слова:** окалина корозійностійких сталей, леговані техногенні відходи, відновна плавка, рентгенофазові дослідження

# STUDYING THE PHYSICAL-CHEMICAL TRANSFORMATIONS AT RESOURCE-SAVING REDUCTION MELTING OF CHROME-NICKEL-CONTAINING METALLURGICAL WASTE

**A. Petryshchev**

PhD, Associate Professor

Department of Labour and Environment Protection  
Zaporizhzhya National Technical University  
Zhukovskoho str., 64, Zaporizhzhya, Ukraine, 69063  
E-mail: kafedrales@ukr.net

**D. Milko**

Doctor of Technical Science, Associate Professor  
Department of Technical Systems of Livestock Technologies  
Tavria State Agrotechnological University  
B. Khmelnytskoho ave., 18, Melitopol, Ukraine, 72310

**V. Borysov**

Head of Laboratory\*

**B. Tsybalya**

PhD

Department of Occupational, Technogenic and Environmental Safety  
National University of Civil Defence of Ukraine  
Chernyshevskaya str., 94, Kharkiv, Ukraine, 61023

**I. Hevko**

Senior Researcher\*

**S. Borysova**

Senior Researcher\*

**A. Semenchuk**

PhD

Department of Applied Mathematics  
Ivano-Frankivsk National Technical University of Oil and Gas  
Karpatska str., 15, Ivano-Frankivsk, Ukraine, 76019  
\*Research Laboratory of Applied Materials Science  
Donbass Institute of Technique and Management Private  
Higher Educational Establishment "Academician Yuriy Bugay  
Internationalscientific and Technical University"  
Mashynobudivnykiv blvd., 32, Kramatorsk, Ukraine, 84313

## 1. Introduction

Large volumes of industrial waste and secondary raw materials in practice are not very effective. Waste doped cor-

rosion-resistant steels, the operation of which may be accompanied by the effects of hostile environments, temperature, and mechanical factors [1], contain high-value elements such as Ni and Cr. An essential part is oxide and fine dispersion

(scale, grinding dust), which is difficult to process efficiently, which adversely affects the manufacturability of production and the cost of production.

Therefore, the problem is to reduce the losses of Ni and Cr in the processing of waste corrosion resistant steels. For this purpose, it is necessary to study the peculiarities of the physical-chemical properties of the scale of corrosion-resistant steels and transformations in the reduction of oxide chromium-nickel-containing metallurgical waste.

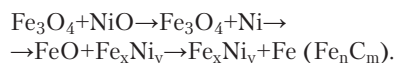
## 2. Literature review and problem statement

Renewable smelting with the use of a carbon reducing agent is one of the technologically simple and economically attractive methods for the processing of technogenic waste. In this case, the presence of residual carbon in the oxycarbide and carbide compounds is possible [2]. But certain allowable carbon surplus provides additional protection against the oxidation of alloying elements and restores residual oxides in a liquid bath while using a doping additive.

According to the results of authors studies [3] the iron scale consists of  $\text{Fe}_3\text{O}_4$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{FeO}$ . Similar results were obtained by authors [4] in the study of rolling iron scale. The possibility of metal phase presence of Fe in the scale of oxide phases is indicated by scientists [5]. According to the results from [6], the phase composition of alloyed Ni (21.99 % by mass) and other elements of the precision alloy scale also consists of iron oxides. This may be related to partial substitution in the oxides with the participation of the atoms of the alloying elements and iron.

Based on this work [3],  $\text{Fe}_3\text{C}$  and C were discovered after carbon black iron oxide together with Fe in reduction products. Similar results were found by authors [7] when carbon-thermal reduction of the scale of chromium-nickel steel. According to the results of phase analysis, the alloying elements were present not in the form of individual compounds, but as a solid solution in the phase of Fe. These technological aspects should be taken into consideration when developing the parameters of regenerative melting of oxide wastes of corrosion-resistant steels. But in these works, the study of the O/C influence in the charge on the phase composition and microstructure of the reduction products is not given.

The authors of work [8] conducted a study into reducing reactions involving oxides and carbon in the Fe–Ni–O system at temperatures up to 1,373 K. With an increase in the temperature of treatment, the reduction products appeared in the following sequence:



That is, there was a relatively greater propensity for the reduction of Ni oxide than Fe oxides. At some stages, the formation of metal Ni and Fe was observed. The authors indicate the possibility of the presence of Fe carbides and iron-nickel-containing phase of  $\text{Fe}_x\text{Ni}_y$ , which may also be manifested in the reduction of oxide waste of corrosion-resistant steels. But in the consideration of the work there is no possibility to follow the course of transformations involving the chromium-containing oxide component, which may be present in the scale of corrosion-resistant steels.

The authors of work [9] investigated the carbon reduction of nickel-iron-containing lateritic ores. After processing

for 60 minutes. At 1,623 K and a molar C/O ratio of 1.4, the degree of removal of Ni and Fe was 96.6 % and 97.9 %, respectively. In this case, the content of Ni and Fe in the product was 9.4 % and 87.5 %, respectively. Similar studies with lateritic ores were carried out by authors of work [10]. Processing at 1,773 K for 90 minutes, and a C/O 1.0 ratio yielded a product of 8.33 % by weight Ni and 84.71 % wt. Fe. That is, it is practically confirmed that the nickel-iron oxide raw material can be restored by a carbon reducing agent with the achievement of a relatively high degree of extraction of target elements. At the same time, the results of C/O ratio studies in the charge on the nature of reduction, which determines the residual carbon content in the products of reduction, are practically significant. But the presence of concomitant ore impurities, in contrast to technogenic oxide raw materials of corrosion resistant steels, can cause significant differences in the physical-chemical characteristics of the reduction and composition of the target product.

Authors of work [11] investigated the reduction of Ni and Fe by carbon from the oxide component of nickel-containing slags. The increase in the processing temperature from 1,373 K to 1,523 K provided an improvement in the degree of reduction (%) of Ni from 66 to 90, Fe – from 20 to 43. The optimum treatment was noted at 1,473 K for 20 minutes with a carbon content of 5 % by weight, which needs to be taken into consideration when developing parameters for recycling waste from corrosion resistant steels. But from the results of this work there is no possibility to follow the change in the microstructure and the composition of individual inclusions and phases of the reduction products when adjusting the processing parameters.

The reduction of  $\text{FeO}\cdot\text{Cr}_2\text{O}_3$  oxides at various C/Fe ratios and temperatures from 1,373 K to 1,523 K was investigated by authors of [12]. They determined that with increasing C/Fe from 0.8 to 1.4, the degree of extraction (%) Cr increased from 9.6 to 74.3, respectively. An increase in temperature to 1,523 K led to an increase in the formation of carbides. The scientists observed a significant reduction at C/Fe below 0.8, in the degree of Cr removal and a decrease in carbide formation. The resulting Cr carbides were dissolved in the Fe phase. It turns out that in order to achieve an increase in the degree of removal of Cr in the reduction of the charge, it requires some excess of carbon relative to oxygen.

Research [13, 14] shows the study of the carbon  $\text{Cr}_2\text{O}_3$  reduction in the range 1,273–1,773 K. A parallel regeneration and formation of  $\text{Cr}_3\text{C}_2$ ,  $\text{Cr}_7\text{C}_3$ ,  $\text{Cr}_{23}\text{C}_6$  carbides was found, while the possibility of obtaining iron-chromic ligatures with a limited carbon content was found. It is possible to obtain products of carbonaceous reduction with relatively low residual carbon content, which testifies to expanded areas of use, including doping of corrosion resistant steels.

It should be noted the presence of significant results of studying the composition of the scale of uncontested grades of steels, presented in works [3–5]. In addition, there are results of phase studies of nickel-containing scale of precision alloy [6]. But at the same time insufficiently studied the physical and chemical properties of the scale of corrosion-resistant chromium-nickel-containing steels. Analysis of the sources of information points to the significant results of studies on the reduction of carbon Ni [8–11] and Cr [12–14] by the example of the individual oxides taken together with the iron oxides. Comprehensive doping can have a significant effect on the reduction process and the final chemical

and phase composition, microstructure of the target product. Some results of carbon monoxide oxidation of corrosion resistant steels are presented by authors of work [7]. But according to the results of this work, it is not possible to follow the structural changes after the reduction of the waste of waste, as solid-phase reduction is investigated. In addition, the effect of O/C change in charge on the phase composition and structure of reduction products is not shown. Research in this direction can reduce the losses of Cr and Ni by sublimation of oxide compounds during the receipt and use of the restored doping material.

Therefore, it is expedient to study the physical and chemical properties of the scale of chromium-nickel-containing steels and the laws of the influence of the parameters of the charge on the phase composition and microstructure of the resource-saving doping raw material obtained by the reduction of smelting. This will allow you to determine the nature of the presence of elements in the material. However, the use of X-ray microanalysis of individual inclusions and phases will provide an expansion of the representation of the distribution of alloying elements in the resulting alloy.

### 3. The aim and objectives of the study

The purpose of this work was to study the physical-chemical characteristics of chromium-nickel-containing wastes from the production of corrosion-resistant steels and alloying, obtained by means of regenerative melting. This is necessary to determine the parameters that reduce the losses of Ni and Cr by sublimation during the processing of oxide doping raw materials and using the resulting dopant.

To achieve this goal, the following tasks were set:

- to determine the peculiarities of the phase composition of the doped alloy based on chrome-nickel-containing technogenic wastes at different ratios of O/C in the charge regarding the nature of the presence of elements;
- to study the microstructure and chemical composition of the scale of corrosion-resistant steels and separate phases and inclusions of a resource-saving chromium-nickel-containing alloy with different ratios of O/C in the charge.

### 4. Materials and methods to study the properties of technogenic raw materials and the resulting alloy

#### 4. 1. Examined materials and equipment used in the experiment

The raw material is a mixture of chromium-nickel-containing corrosion-resistant steels scale of type 18–10, which was formed on redistribution of metallurgical production. The reducing agent is carbon, in the form of ultrafine dust from carbon-forming production (the share of carbon is 98 % by weight), the amount of which regulates the change in the ratio of O/C in the charge in the range of 1.09–1.78. For the intensification of the heating of the charge and the reduction processes, the grinding of the steel 10X17H13M2T was added. Samples for research were smelted in an indirect furnace heater with coal lining in alundum crucibles. Melting point is 1,873–1913 K. After smelting, the alundum crucibles, together with the alloy, were removed from the furnace and cooled at ambient temperature.

X-ray diffraction analysis of samples was carried out at the diffractometer “DRON-6” (Russia).

Photographs of the microstructure of the samples were obtained from the raster electron microscope “REM-106I” (Ukraine). The microscope is equipped with a system of X-ray microanalysis with the determination of the chemical composition of individual areas of the surface of the samples.

#### 4. 2. Methods for conducting experiments and determination of properties samples indicators

The phase composition of the samples was determined by X-ray diffraction analysis using monochromatic radiation of  $\text{CuK}\alpha$  ( $\lambda=1.54051 \text{ \AA}$ ) from the Ni filter. The measurements were carried out at a voltage of  $U=40 \text{ kV}$  and an anode current  $I=20 \text{ mA}$ . The phase composition was determined using the PDWin 2.0 software suite (Russia).

Investigation of the microstructure of the samples was performed at an accelerating voltage of 20 kV and a current of an electron probe of 52–96  $\mu\text{A}$ . The working distance to the investigated surface was 10.7–11.6 mm. Determination of the composition phases performed without the standard method of calculating the fundamental parameters.

### 5. Results of research into properties of the source of technogenic raw material and the resulting doped alloy

#### 5. 1. Determination of the peculiarities of the alloy phase composition with different O/C in the charge

The phase composition of the O / C alloy in the charge of 1.78 consisted mainly of  $\gamma\text{-Fe}$  and  $\text{Fe}_3\text{C}$ , whose manifestation was characterized by relatively low intensity (Fig. 1). At O/C in the charge 1.33 there is a slight decrease in the manifestation of  $\gamma\text{-Fe}$  and an increase in the intensity of the diffraction peaks  $\text{Fe}_3\text{C}$ .

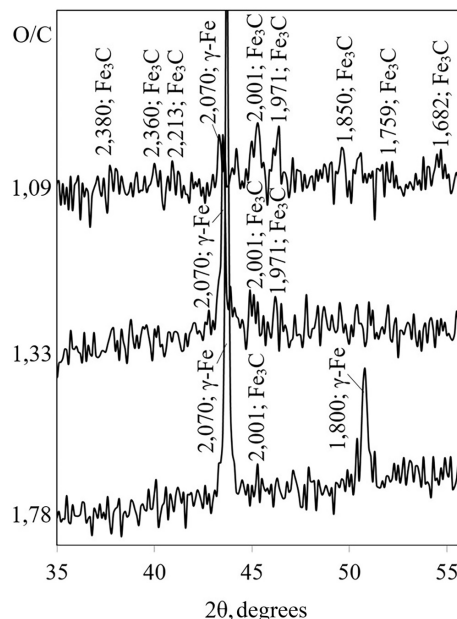


Fig. 1. Fragments of the doped alloy diffractograms at a different ratio of O C in the charge

The ratio of O/C in the charge at 1.09 provided the predominant  $\text{Fe}_3\text{C}$  in the alloy with a relatively low  $\gamma\text{-Fe}$  mapping intensity. The presence of the individual compounds of the alloying elements may also be present, but they have

a fragmentary nature of the diffraction peaks at relatively weak intensity.

**5. 2. Investigation of the microstructure of the initial scale and the doped alloy with different O/C in the charge**

The microstructure of a scale mixture of chromium-nickel-containing corrosion-resistant steels is disordered with the presence of different sizes particles and shapes (Fig. 2). The chemical composition of the studied scale is given in Table 1.

Table 1

Chemical composition of the examined section of scale is, respectively, Fig. 1

| Element content, % wt. |       |      |      |      |      |      |      |       |               |
|------------------------|-------|------|------|------|------|------|------|-------|---------------|
| Fe                     | Cr    | Ni   | Mo   | Ti   | Ca   | Si   | Al   | O     | Total content |
| 43.64                  | 14.26 | 7.65 | 0.51 | 0.84 | 1.03 | 1.55 | 0.82 | 29.70 | 100           |

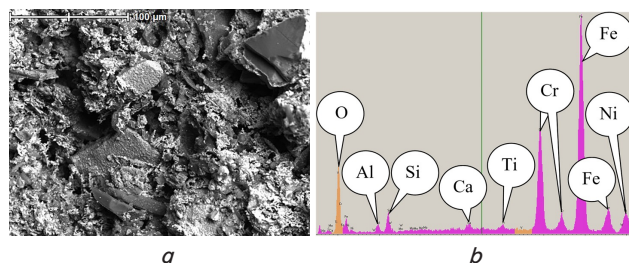
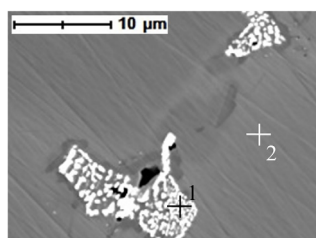
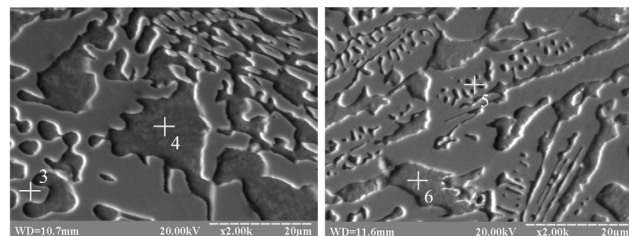


Fig. 2. Microstructure plot with X-ray microanalysis of a chromium-nickel-containing corrosion-resistant steels scale mixture : *a* – photograph at magnification  $\times 500$ ; *b* – spectrograph of X-ray microanalysis

The microstructure of the investigated alloy consisted of several phases (Fig. 3) with different shape and particle size. At O/C in the charge of 1.78 there is a vast amount of phase with a relatively high Ni content of 15.16 % by weight (Table 2, Fig. 4) and Cr content 2.64 % by weight.



*a*



*b*

*c*

Fig. 3. Microstructure of the doped alloy obtained with different ratio of O/C in the charge and increase: *a* – 1.78,  $\times 3,000$  (sample without etching), *b* – 1.33,  $\times 2,000$  (etching sample), *c* – 1.09,  $\times 2,000$  (etched sample)

Table 2

Results of X-ray microanalysis of doped alloy in accordance with Fig. 3

| Research points composition of samples | Element content, % wt. |       |       |      |      |      |      | Total content |
|--|------------------------|-------|-------|------|------|------|------|---------------|
|  | Fe                     | Cr    | Ni    | Mo   | Ti   | S    | P    |               |
| 1                                      | 64.06                  | 32.90 | 1.41  | 1.30 | 0.33 | 0.00 | 0.00 | 100           |
| 2                                      | 81.24                  | 2.64  | 15.16 | 0.85 | 0.11 | 0.00 | 0.00 | 100           |
| 3                                      | 76.89                  | 17.74 | 4.15  | 1.03 | 0.19 | 0.00 | 0.00 | 100           |
| 4                                      | 79.71                  | 2.79  | 16.94 | 0.38 | 0.17 | 0.01 | 0.00 | 100           |
| 5                                      | 85.45                  | 8.25  | 4.93  | 1.07 | 0.30 | 0.00 | 0.00 | 100           |
| 6                                      | 76.60                  | 1.27  | 20.90 | 1.15 | 0.07 | 0.01 | 0.00 | 100           |

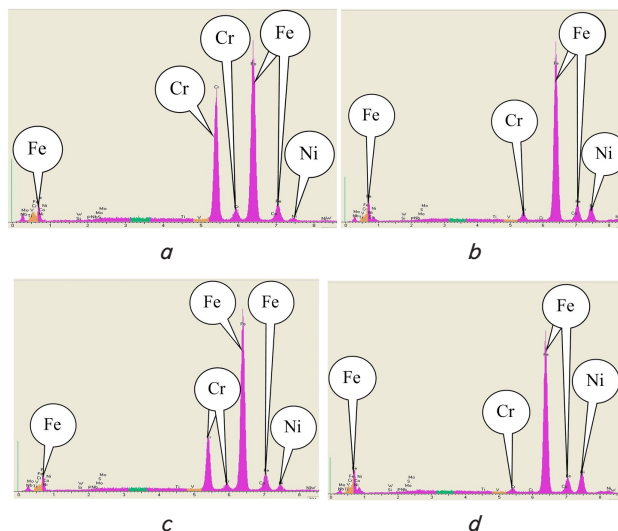


Fig. 4. Spectrographs of some of the examined points in Fig. 3: *a* – 1, *b* – 2, *c* – 3, *d* – 6

Along with this, we detected the presence of individual particles of the phase with an elevated Cr content of 32.90 % by weight with a content of Ni – 1.41 % by weight.

At O/C in the charge 1.33 and 1.09, photographs of the microstructure of the alloy showed an increase in the amount of phase with a relatively high Cr content (Fig. 3, points 3, 5). The Cr content in this phase was, respectively, 17.74 % by weight and 8.25 % by weight at a Ni content of 4.15 % by weight, respectively, and 4.93 % by weight.

The phase with a relatively high content of Ni (16.94 % by weight and 20.90 % by weight at O/C in the charge of 1.33 and 1.09, respectively) was more affected by poisoning. The amount of Mo and Ti in the investigated sites was within the range of 0.38 to 1.30 % by weight and 0.11–0.33 % by weight, respectively (Table 2). The presence of P was not detected. The content of S did not exceed 0.01 % by weight.

**6. Discussion of results of studying the properties of technogenic raw materials and the resulting doped alloy**

The conducted studies indicate that the initial mixture of chromium-nickel-containing corrosion-resistant scale steels has an increased alloying of Ni – 7.65 % by weight and Cr – 14.26 % by weight. The ordering of the investigated of the scale microstructure (Fig. 2) indicates the possibility of

uneven distribution of the alloying elements and the different phase composition in separate particles. Content of O at 29.70 % by weight indicates that Fe and alloying elements may be in the scale in the form of an oxide component. This fits well with the results of works [3–5], where the phase composition of the iron scale is represented by  $\text{Fe}_3\text{O}_4$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{FeO}$ . The alloying elements are likely to be present, to some extent, as substitution atoms in iron oxides. This pattern was a manifestation in the case of Ni atoms in the results of work [6] when studying the phase composition of alloyed scale of a precision alloy. However, it is possible that some of the alloying elements may be present as separate oxide compounds.

The phase analysis of the alloy with different O/C in the charge (Fig. 1) indicates that the alloying elements are in a solid solution in the  $\gamma$ -Fe lattice. Some of the alloying elements may be as substitution atoms in a  $\text{Fe}_3\text{C}$  compound. This is much in the line with the results of work [7], where only the iron-containing compounds were clearly manifested in the diffractogram of the restored doped product. Relatively weak manifestation of the presence of  $\text{Fe}_3\text{S}$  carbide was observed even with a lack of C (O/C=1.78). This confirms the impossibility of obtaining a fully carbon-free product and it fits with the results of work [2]. A further change in O/C in the charge of 1.33 and 1.09 caused an increase in  $\text{Fe}_3\text{C}$ .

The presence of the atoms of the alloying elements in the  $\gamma$ -Fe lattice and the  $\text{Fe}_3\text{C}$  carbides indicates the results of the microstructure study and X-ray microanalysis (Fig. 3, 4, Table 2). It is evident that some phases have an elevated Cr content in Fe (Table 2, points 1, 3, 5). These phases, probably, may consist of  $\text{Fe}_3\text{C}$  carbide, where Fe atoms are partially replaced by Cr atoms. This is consistent with the results of work [13, 14], which indicates the formation of chromium-containing carbides in parallel with the reduction. The possibility of dissolving a certain fraction of carbides in  $\gamma$ -Fe at the contact of two phases is not excluded, which is also indicated by authors of work [12].

The phase with relatively high Ni content (Table 1, points 2, 4, 6) is probably found to be a solid solution of alloying elements in  $\gamma$ -Fe in the microstructure photographs. This is much in the line with the results of work [8–11], in which the relatively high efficiency of the reduction of the iron-nickel oxide component with carbon is confirmed.

The presence of P was not detected. The content of S is not more than 0.01 % by weight, which is lower than the maximum permissible concentration in the target for alloying of stamps. In the obtained samples there are no compounds and phases with a relatively high susceptibility to sublimation. There is no need to create special conditions that prevent the evaporation and loss of alloying elements with the gas phase. It also causes an increase in the use of alloying elements.

Based on the results of our study, it follows that the most acceptable ratio of O/C in the charge is 1.78. In this case, the reduction with a predominance in the phase composition  $\gamma$ -Fe with a relatively weak manifestation of residual carbon, as a carbide component, is provided. We determined the indicators for obtaining the product of remediation of manmade chrome-nickel-containing waste products with relatively low carbon content. This expands the possibility

of using the resulting alloy with the replacement of standard alloying materials portion of standard alloying materials when smelting grades of steel with restrictions on carbon content.

It is possible to note as a shortcoming the absence of results of physical and chemical properties research of separate particles and inclusions of microstructure of initial scale. This would enable a more detailed study of the nature of the presence of elements in the disordered microstructure of doped oxide raw materials.

The development of this study is possible in the direction of expanding the range of grades of steels, whose oxide waste will be involved in the processing by the method of reducing melting. The promising wastes with an increased level of doping.

Experimental-industrial melting of corrosion-resistant steel of type 18–10 in the arc furnace DSW-40 with the main lining using doped alloy on the basis of technogenic wastes was made. The alloy corresponded to the composition, % by weight: C – 0.03–0.05, Si – 0.50–1.80, Mn – 0.5–2.0, Cr – 15.0–25.0, Ni – 8.0–14.0, Ti – 0.50–1.20, Al – 0.50–1.80,  $\text{Cu} \leq 0.50$ ,  $\text{S} \leq 0.025$ ,  $\text{P} \leq 0.035$ , Fe – residue. The doped alloy was used as a bilayer billet with a consumption coefficient of 270–330 kg/ton of steel as a partial replacement of standard alloying materials. The use of a doping alloy provided a reduction in the consumption of Ni and Cr by 27–31 % by weight, and 25–29 % by weight, respectively. Parching of alloying elements decreased by 3–4 % by weight.

## 7. Conclusions

1. We determined that in the alloy at a ratio of O/C in the charge in the range of 1.09–1.78 there are  $\gamma$ -Fe and  $\text{Fe}_3\text{C}$  phases with doping elements as substitution atoms. At O/C=1.78, the phase composition consisted predominantly of  $\gamma$ -Fe in a weak  $\text{Fe}_3\text{C}$  expression. The phase change of O/C in the charge at 1.33 and 1.09 resulted in an increase in  $\text{Fe}_3\text{C}$  in diffractograms. The presence of some of the individual compounds of alloying elements is not excluded, but they have a fragmentary nature of the diffraction peaks with relatively weak intensity.

2. We determined that the microstructure of a scale mixture of chromium-nickel-containing corrosion-resistant steels is disordered with the presence of particles of different sizes and shapes. The content of alloying elements Ni and Cr was 7.65 % by weight and 14.26 % by weight, respectively, at an oxygen content of 29.70 % by weight. In a relatively small amount in scale, Mo and Ti were found to be 0.51 % by weight and 0.84 % by weight, respectively. The microstructure of the doped alloy with different O/C in the charge had a clear manifestation of several phases, characterized by a difference in the content of the main alloying elements. The content of Ni in the investigated sections of different phases varied within the range of 1.41–20.90 % by weight, Cr – 1.27–32.90 % by weight. The presence of Mo and Ti was within the range of 0.38 to 1.30 % by weight and 0.07–0.33 % by weight, accordingly. The presence of P was not detected. The content of S did not exceed 0.01 % by weight.

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